PATENT SPECIFICATION

NO DRAWINGS

875,133

Date of Application and filing Complete Specification Nov. 14, 1957.

No. 35552/57.

Application made in Italy on Nov. 15, 1956.

Complete Specification Published Aug. 16, 1961.

Index at acceptance:—Classes 2(6), P7(A:C10:D1X:S2); 2(2), B2B2.

International Classification:—C08f. D01f.

COMPLETE SPECIFICATION

Preparation of Uniformly Dyed Elongated Articles of Polypropylene

We, MONTECATINI SOCIETA GENERALE PER L'INDUSTRIA MINERARIA E CHIMICA, a Body Corporate organised and existing under the laws of Italy, of 18, Via Filippo Turati, Milan, Italy, do hereby declare this invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the preparation of uniformly dyed or coloured elongated articles formed predominantly from highly crystalline polypropylene, that is to say articles such as yarns, fibres, tapes and films formed predominantly of highly crystalline polypropylene which have one or both of their transverse dimensions small compared with their length dimensions. Such articles may be produced for example by extrusion or spinning.

It is known that highly crystalline polypropylene is a colourless material with a high melting point and insoluble in the common solvents, which although it can be formed into elongated articles having very useful physical, chemical and mechanical properties, has a very low affinity for dyes.

We have now found that, in the case of polypropylene, it is possible to obtain uniformly dyed manufactured articles by having recourse to a bulk dyeing process.

The invention provides a process for the preparation of uniformly dyed elongated articles formed predominantly of highly crystalline polypropylene which process comprises mixing at least one dye which is stable at the melting point of the polypropylene and does not effect a degrading action thereon, with the polypropylene, melting or dissolving the polypropylene and subsequently forming the elongated articles.

The article may be formed by an extrusion or spinning process and either organic dyes or inorganic pigments may be used.

The dye may be mixed with polypropylene in any of several ways:—

1) It can be introduced before the polypropylene is melted, by mixing it intimately with polypropylene in powdered form.

2) It can be introduced into the polypropylene after the polypropylene has been melted or is in the form of a solution or

3) The dye and polypropylene may be dissolved in a common solvent.

4) The dye and polypropylene may be dissolved in a mixture of two or more solvents at least one of which is a solvent for the dye.

Suitable organic dyes are anthraquinone, aminoanthraquinone, indigo, thioindigo, phthalocyanine and azo dyes. In general, both organic dyes and inorganic pigments used must have the following essential properties, namely, stability at the melting point of polypropylene and a non-degrading action towards the polypropylene.

Mixtures of various dyes may be used to obtain particular dyeing effects provided that each component has these properties.

The dyed articles prepared by the process of this invention show a uniform distribution of the dye (as can be seen from examining thin sections under the microscope), and colours that are fast to light, washing and rubbing. Moreover, the dyes have no or only a very low tendency to migrate towards the polypropylene surface. Such a tendency which is common with many polymers tends considerably to reduce abrasion resistance.

The following examples are given to illustrate the invention:—

EXAMPLE 1

5 g of a powdered dye of the anthraquinone series (Romanthrene yellow GCN) are added to 500 g of finely powdered crystalline poly-

[Price 3s. 6d.]

propylene. The mixture is introduced into a Werner type mixer and mixed for 30 minutes.

The mixture is melted at 250° C. and then extruded through a spinneret having 18 holes of 0.3 mm diameter.

The filaments thus obtained are stretched with a 1:5 ratio on a heated plate. A yarn of very deep and uniform yellow colour is obtained which is subjected to serimetrical tests and gives the following results:—

tenacity 4.6 g/den
elongation 25%.

These data compared with those determined on a filament prepared from a polymer having the same physical characteristics of the test-polymer but without any dye, show that the dye causes no degradation of polypropylene; as a matter of fact the non-dyed yarn shows the following characteristics:—

tenacity 4.7 g/den
elongation 24%.

The colour-fastness tests, carried out according to the specifications of the "International Fastness Code", give the following results:— (dye: Romanthrene yellow GCN; extrusion temperature: 230° C; permanence time: 7 minutes)

natural light	6
washing	5
perspiration	5
fulling	5
trichlorethylene	5

Three other spinning tests were carried out in the same way employing the following dyes:

Indanthren Scarlet 2G - - - - - (anthraquinone series)
Durindone Scarlet YP - - - - - (thioindigo series)
Polymon blue GS - - - - - - (phthalocyanine series)

The colours obtained have good intensity and uniformity and the serimetrical characteristics are the following:—

Indanthren Scarlet 2G - - - - - tenacity 4.92 g/den, elongation 23%
Durindone Scarlet YP - - - - - tenacity 4.9 g/den, elongation 22.5%
Polymon blue GS - - - - - - tenacity 4.89 g/den, elongation 23.5%

The colour-fastness is as follows:—

INDANTHREN SCARLET 2G

fastness to natural light	5
fastness to washing	5
fastness to perspiration	5
fastness to rubbing	5
fastness to fulling	5
fastness to trichlorethylene	5

DURINDONE SCARLET YP

fastness to natural light	5
fastness to washing	5
fastness to perspiration	5
fastness to rubbing	4
fastness to fulling	5
fastness to trichlorethylene	5

POLYMON BLUE GS

fastness to natural light	5
fastness to washing	5
fastness to perspiration	5
fastness to rubbing	5
fastness to fulling	5
fastness to trichlorethylene	5

EXAMPLE 2

A mixture of 500 g powdered crystalline polypropylene and 5 g Romanthrene GCN yellow is intimately mixed in a Werner type mixer for 30 minutes. The mixture is melted and extruded in order to obtain a uniformly dyed film.

The following dyes were also used:—
Indanthrene Scarlet 2G
Durindone Scarlet YP
Polymon blue GS.

The extrusion temperature is 250° C. Film sections are cut from each, which, under the microscope, show no portions of clotted dye and are very transparent.

EXAMPLE 3

A solution is prepared by dispersing in the cold 18 parts by weight of crystalline polypropylene having an intrinsic viscosity of 2.6 (determined in a 1% by weight solution of tetrahydronaphthalene at 135° C.), in 62 parts by weight of a petroleum fraction boiling at 180—200° C. 20 parts by weight cyclohexanone containing 0.36 parts by weight dissolved Oracet B blue are added. The whole is heated to 150° C. while stirring. The mixture is then filtered through a filter-press and extruded through a spinneret having 30 holes of 200μ diameter, into an evaporation tower with circulating air which removes the solvent vapours. Dowtherm (Registered Trade Mark) at 300° is circulated in the tower jacket. The solution is delivered from the spinneret in the form of filaments at a rate of 25 m/minute and after a 6 m run in the evaporation tower, is wound up on a bobbin at a rate of 125 m/min. The yarn obtained is stretched with a 1:5 ratio at 145° C. in warm air. The yarn has the following characteristics:—

tenacity 5.4 g/den
elongation 22%
intensive and uniform colour under the microscope.

Colour fastness:—

to natural light	6/7
to washing	5
to rubbing	5
to perspiration	5

The intrinsic viscosity, determined as previously described on the yarn dissolved in tetrahydronaphthalene, is 2.54.

It will of course be appreciated that the process of the present invention may be, and preferably is carried out in conjunction with one or more of the processes described in several of our copending applications. For example stabilizing treatment as described in Application No. 22342/57 (Serial No. 813,891), filtering procedure as described in Application No. 27758/57 (Serial No. 827,424), cooling procedure as described in Application No. 31404/57 (Serial No. 875,132), the procedure of stretching in the presence of a plasticiser or swelling agent as described in Application No. 33069/57 (Serial No. 817,125), the spreading and volatilisation procedure as described in Application No. 9676/57 (Serial No. 853,637), or the thermal degradation procedure described in Application No. 36349/56 (Serial No. 835,038).

WHAT WE CLAIM IS:—

1. A process for the preparation of uniformly dyed elongated articles formed predominantly of highly crystalline polypropylene which process comprises mixing at least one dye which is stable at the melting point of the polypropylene and does not effect a degrading action thereon, with the polypropylene, melting or dissolving the polypropylene and subsequently forming the elongated articles.

2. A process according to Claim 1, wherein the article is formed by extrusion.

3. A process according to Claim 1, wherein the article is formed by spinning.

4. A process according to any one of Claims 1 to 3, wherein the dye is an organic dye.

5. A process according to any one of Claims 1 to 3, wherein the dye is an inorganic pigment.

6. A process according to any one of the preceding claims, wherein the dye is introduced into the polypropylene in powdered form before the polypropylene is melted.

7. A process according to any one of Claims 1 to 5, wherein the dye is introduced into the polypropylene after the polypropylene has been melted.

8. A process according to any one of Claims 1 to 5, wherein the dye and polypropylene are dissolved in a common solvent.

9. A process according to any one of Claims 1 to 5 in which the dye and the polypropylene are dissolved in a mixture of two or more solvents at least one of which is a solvent for the dye.

10. A process for the preparation of uniformly dyed elongated articles substantially as herein described and illustrated by the foregoing examples.

11. Uniformly dyed elongated articles when prepared by the process claimed in any one of the preceding claims.

ERIC POTTER AND CLARKSON,
Chartered Patent Agents,
317, High Holborn,
London, W.C.1.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1961.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.